



# Development of a Novel Ceramic-to-Metal Seal for High-Temperature, High-Pressure Applications

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# Outline

- Technical challenge: High-T, High-P ceramic-to-metal seals
- Solution: Cermets-filled U-ring seal design concept
- Seal performance goal: 800°C, 100 cycles, 3000 psi
- Experimental
- Results for Ag at 700 °C and AgPd U-rings at 750 °C
- Failure analyses (FA)
- FEA modeling for FA, seal design, and process optimization
- High-T, High-P seal applications and voice of customer
- Summary and Future Efforts

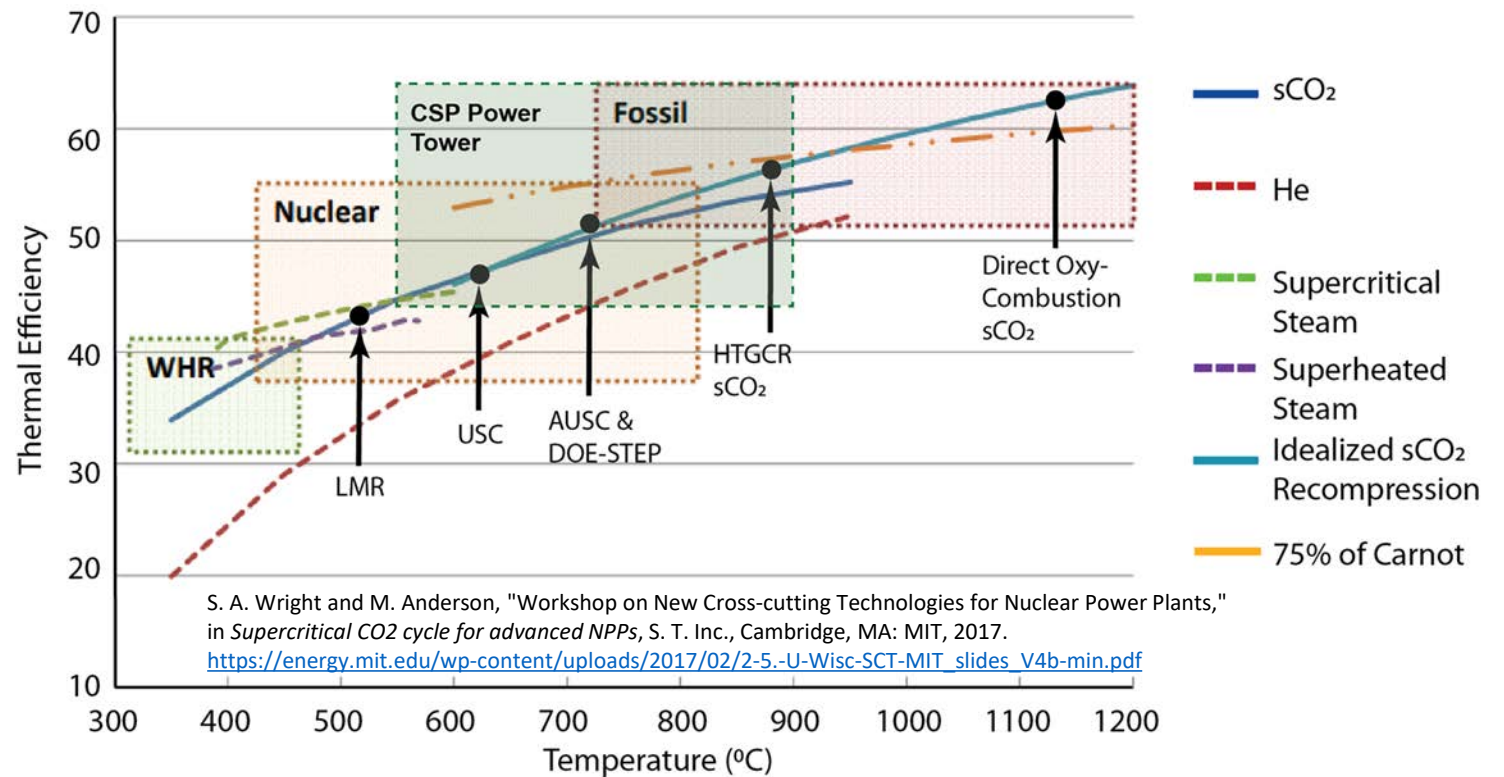
# New High-Temperature, High-Pressure Seals Needed

- Reliable high-temperature seals of metal pipes to ceramic heat exchangers are required
- Enables advanced high-temperature power generation systems that utilize power cycles based on steam or supercritical CO<sub>2</sub> (sCO<sub>2</sub>)



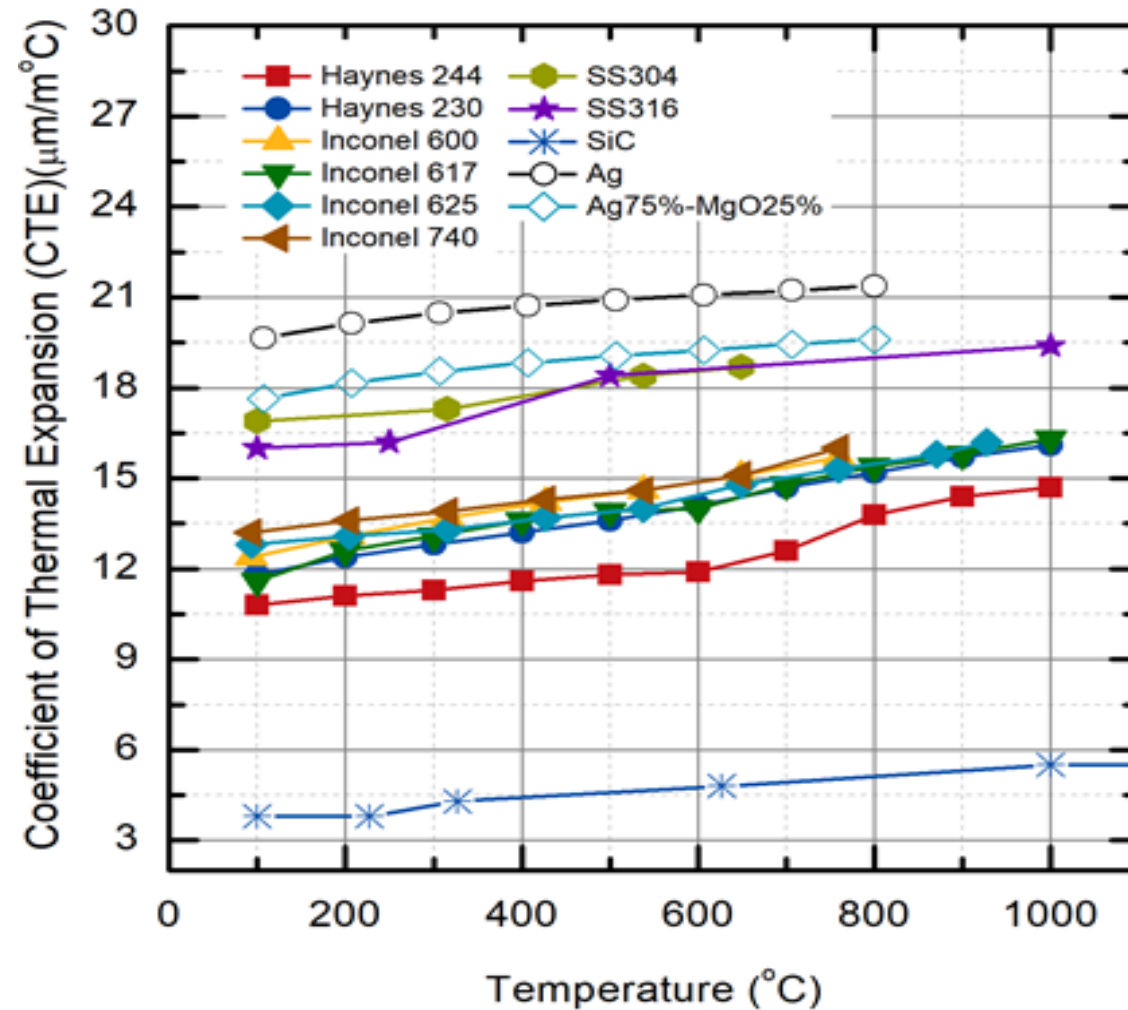
Lewinsohn, Charles. "High-efficiency, ceramic microchannel heat exchangers." *Bull. Am. Ceram. Soc.* 94, no. 5 (2015): 26-31.

Lewinsohn, Charles A., Merrill A. Wilson, Joseph R. Fellows, and Hyrum S. Anderson. "Fabrication and joining of ceramic compact heat exchangers for process integration." *International Journal of Applied Ceramic Technology* 9, no. 4 (2012): 700-711.



# High-Temperature, High-Pressure Seal Challenge

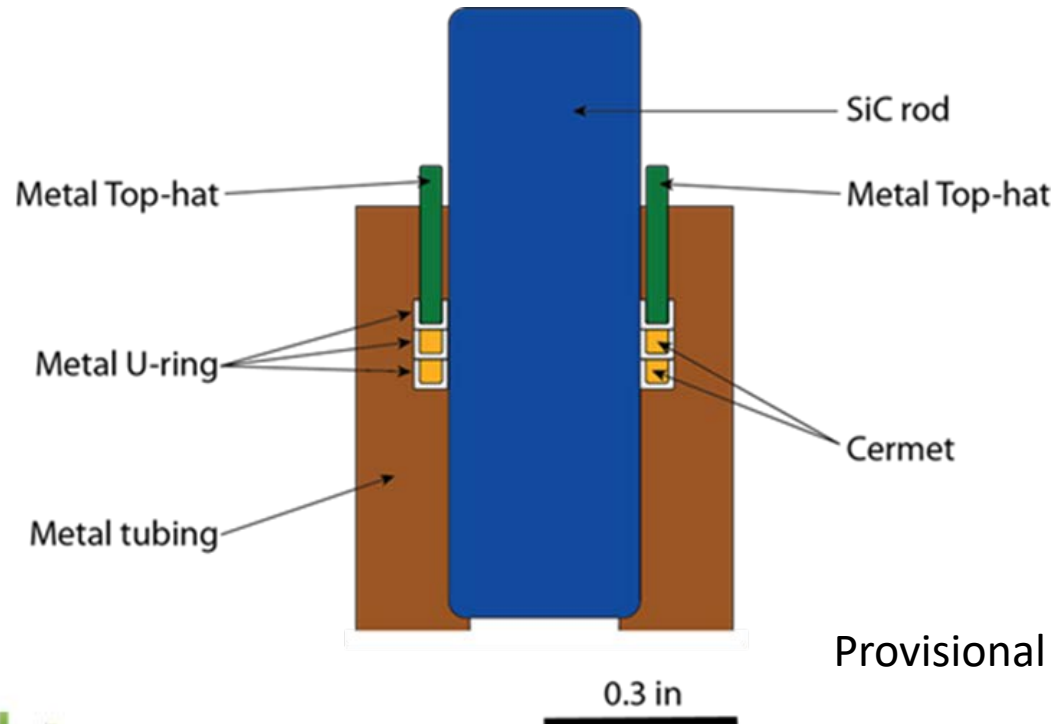
- CTE mismatch between SiC/Si<sub>3</sub>N<sub>4</sub> and metals creates challenges for sealing
- Cermet-filled U-Ring seal formed in situ provides solution



[1] BSU measured the CTE of SiC, Ag, and Ag75%-MgO25%

# Technical Approach

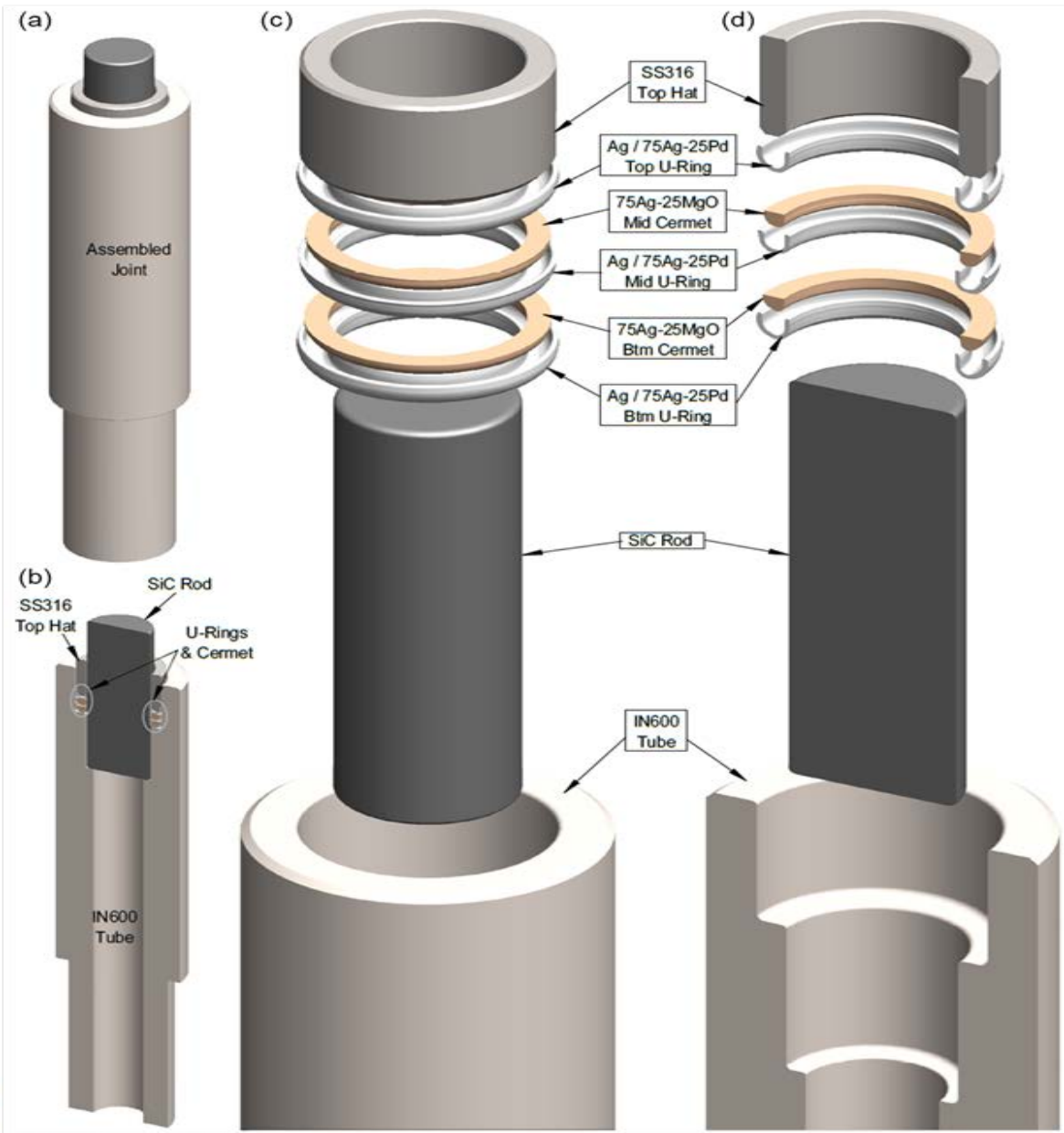
- Cermet-filled U-Ring seal assembly formed in situ at high temperature (maximum gap)
- CTE of cermet powder (Ag/MgO) is higher than SiC or IN600 that are being joined together
- Cermet-filled U-Ring seals are plastically deforming during hot seal forming process
- Seals fill the gap between a metal tube and ceramic rod (tube)
- MgO component of cermet provides stability and strength at high T and P
- Maintains high-pressure gas-tight seal during thermal cycling



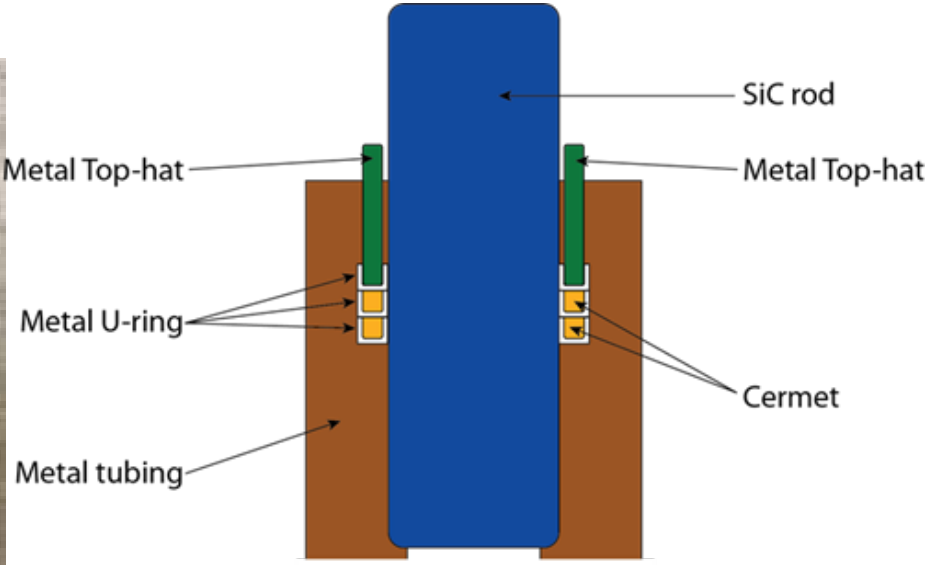
Provisional Patent: 62/728,449



# Cermet-Filled U-Ring Seal Design



Seal test sample



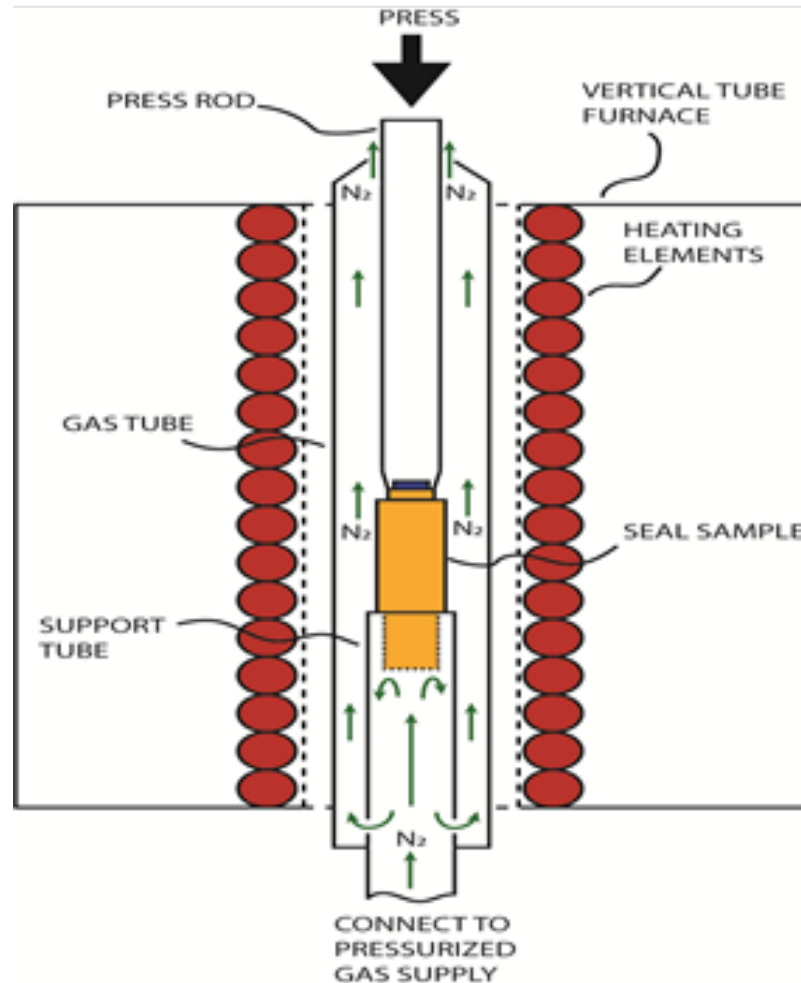
U-Ring (ID=0.315")

0.3 in

# Hot Seal Forming Apparatus



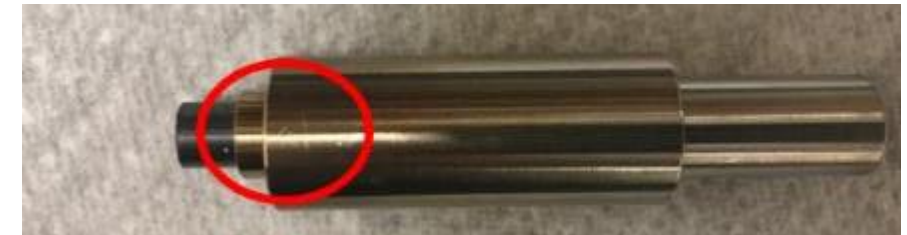
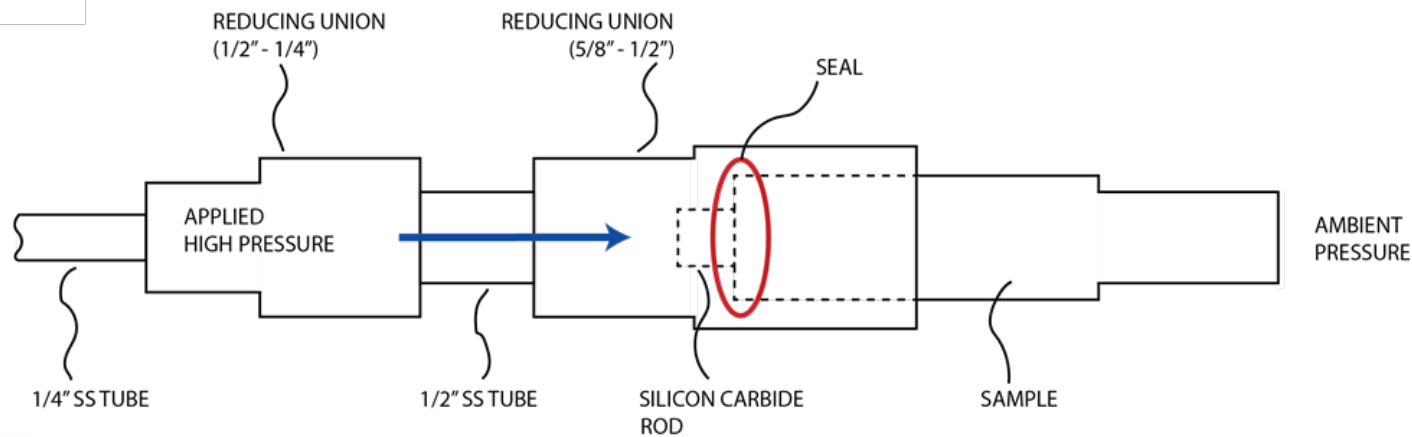
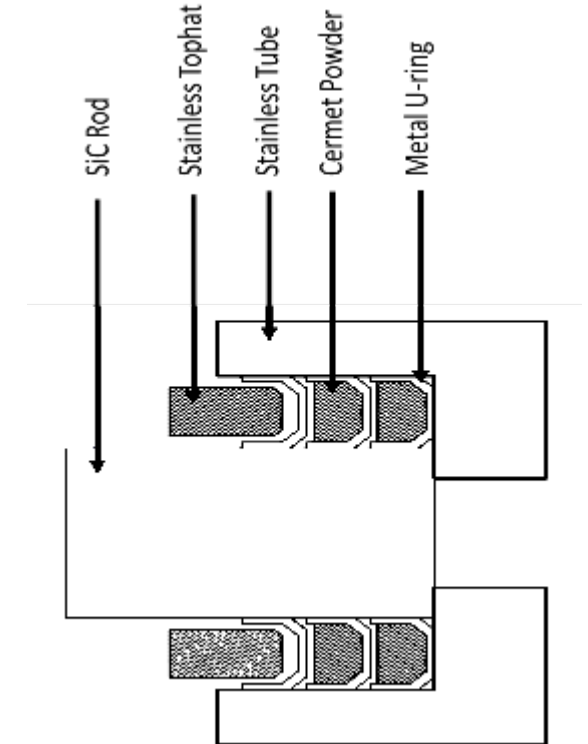
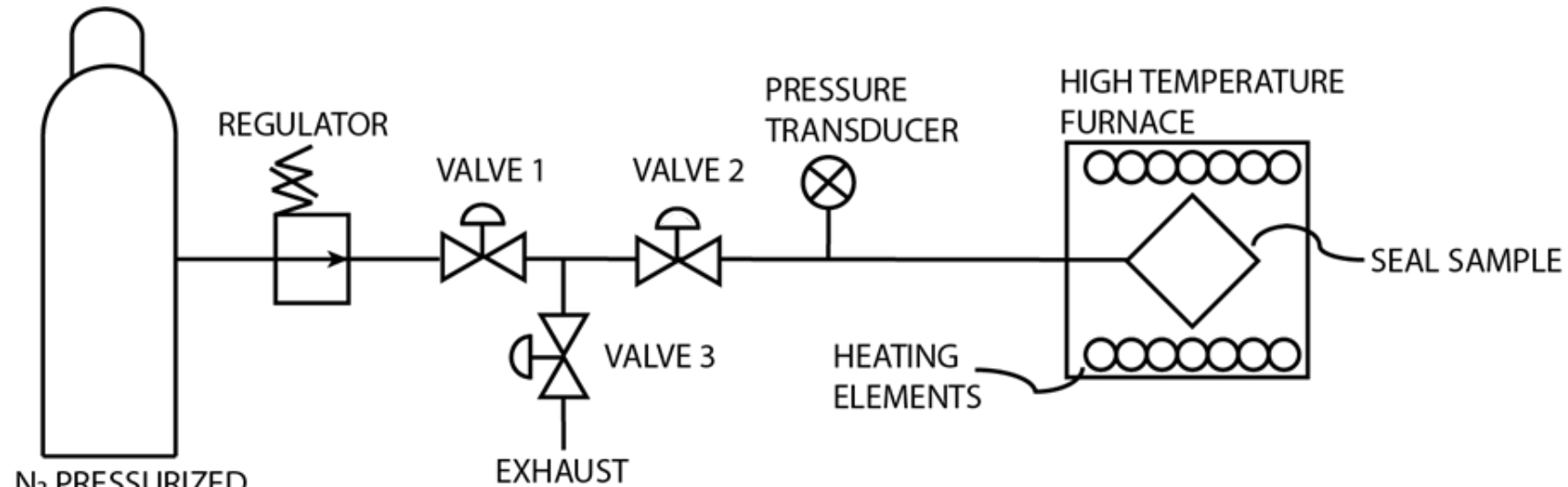
Hot hydraulic press



System schematic

- Seals are assembled and then pressed under N<sub>2</sub> at 900°C and 13,700 psi
- For testing, seals are cycled between atmospheric conditions and high pressure (3000 psi) and high temperature (700-800°C)
- Pressure decay shows seal performance
- After testing, samples are x-ray imaged, cross-sectioned, and characterized

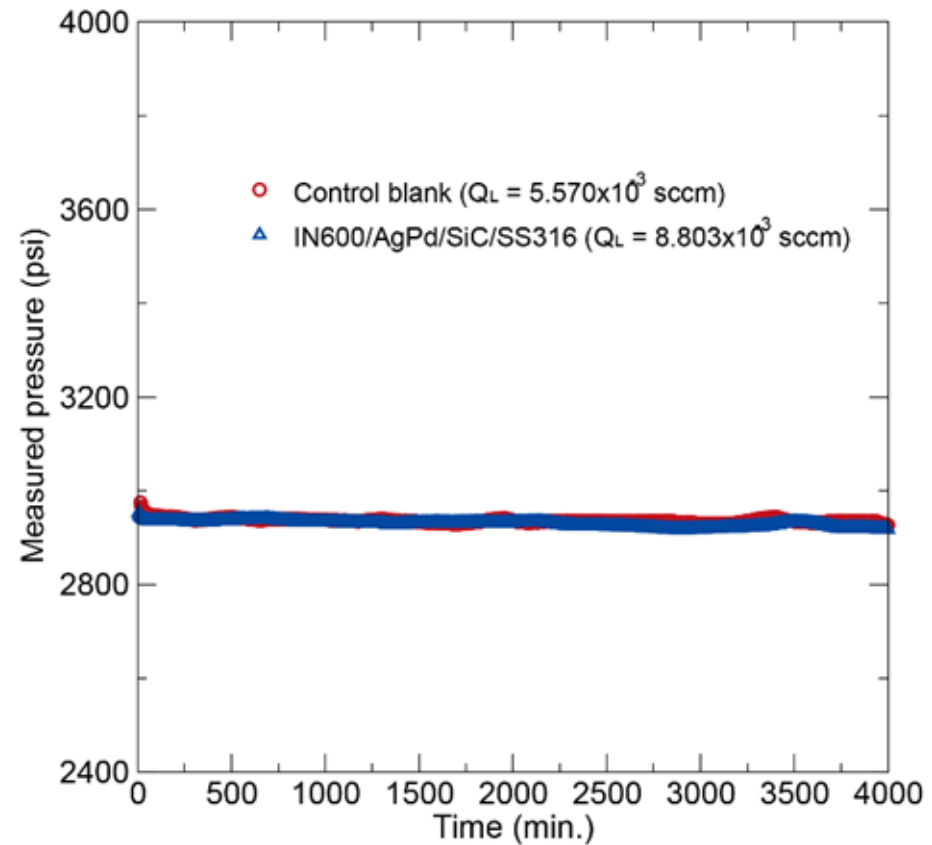
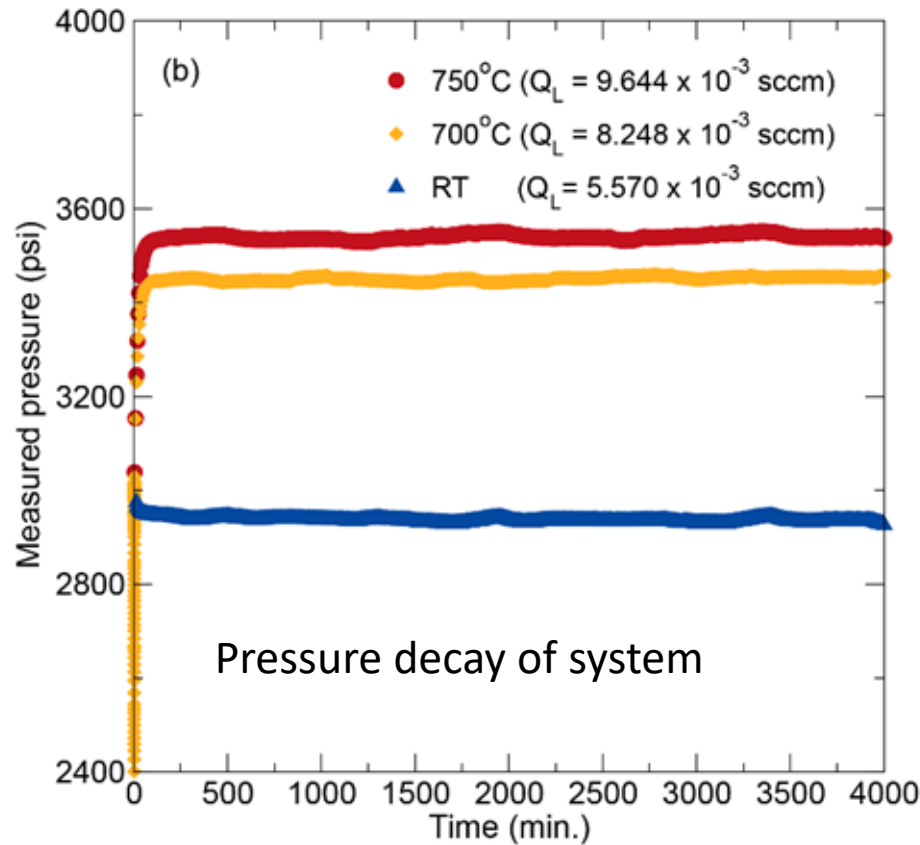
# High Temperature and Pressure Leak Test Rig





# High Temperature and Pressure Leak Test Rig Capability

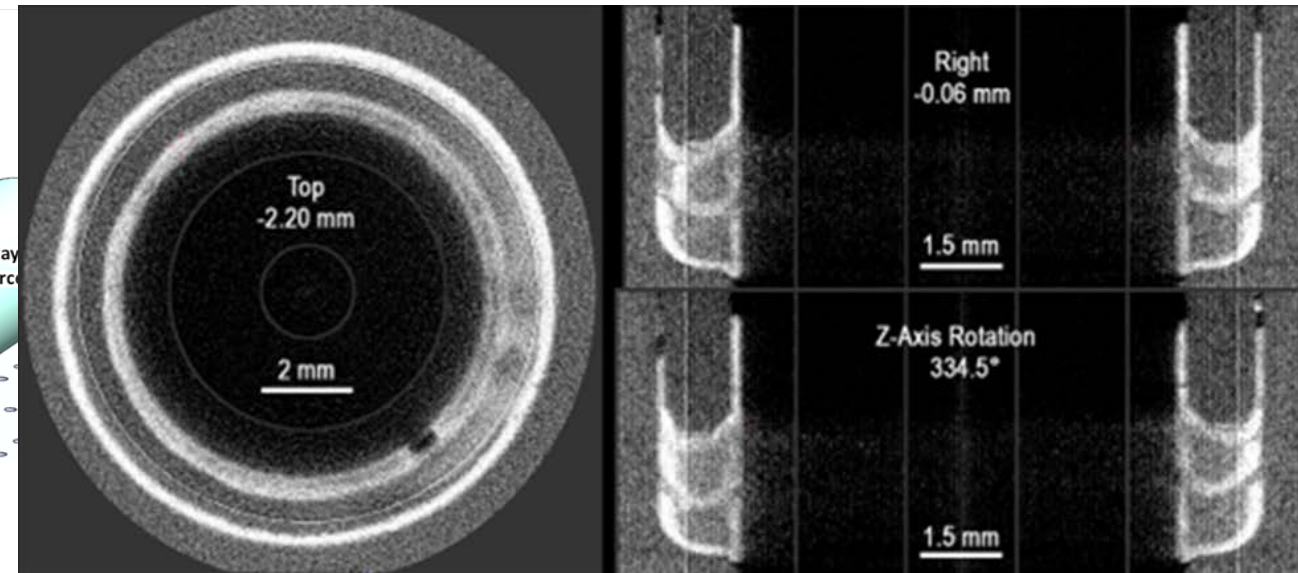
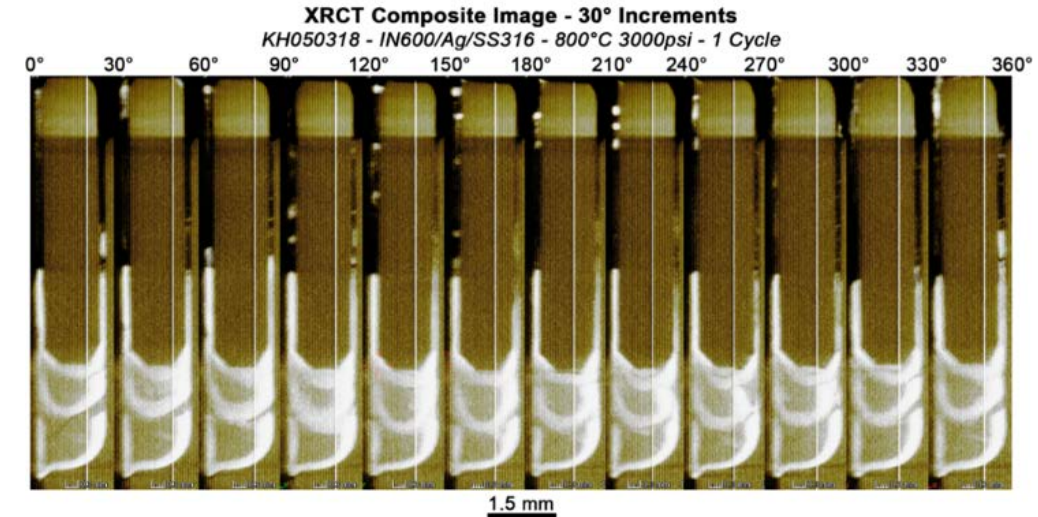
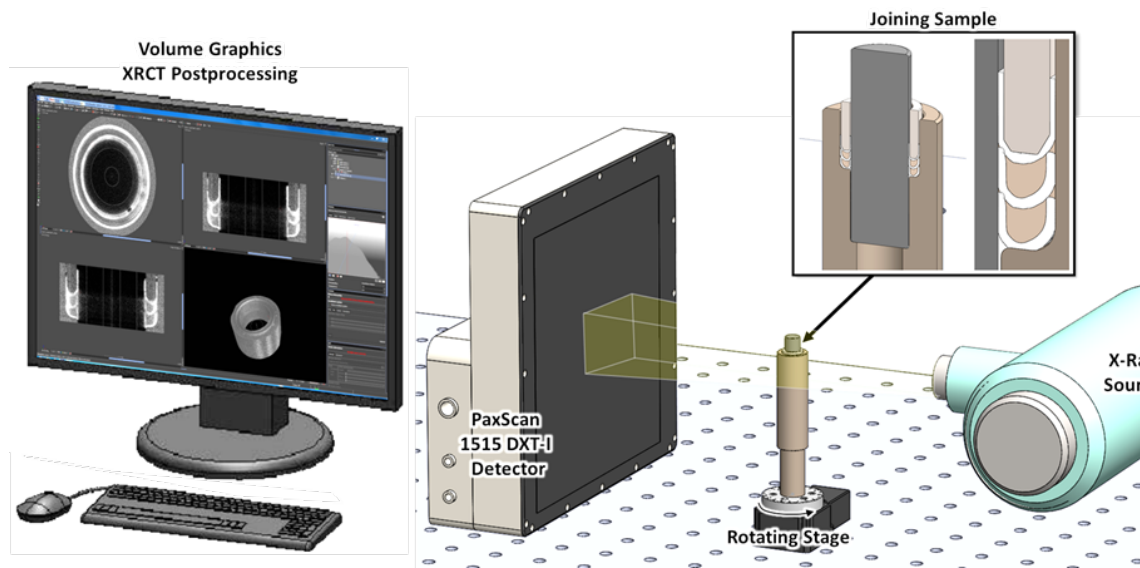
- Pressure decay leak measurement system is capable of detecting small leaks (<0.01 sccm) at elevated temperatures and pressures
- Trade-off between measurement time and leak rate measurement accuracy



# Seal Failure Analyses: X-Ray Computed Tomography (XRCT)

- Collaborating with INL<sup>1</sup> to perform XRCT scans of failed and control seal assemblies
- XRCT to nondestructively observe seals
- Cross section at specific locations to better understand chemistry and microstructure

*XRCT Characterization Overview Schematic*

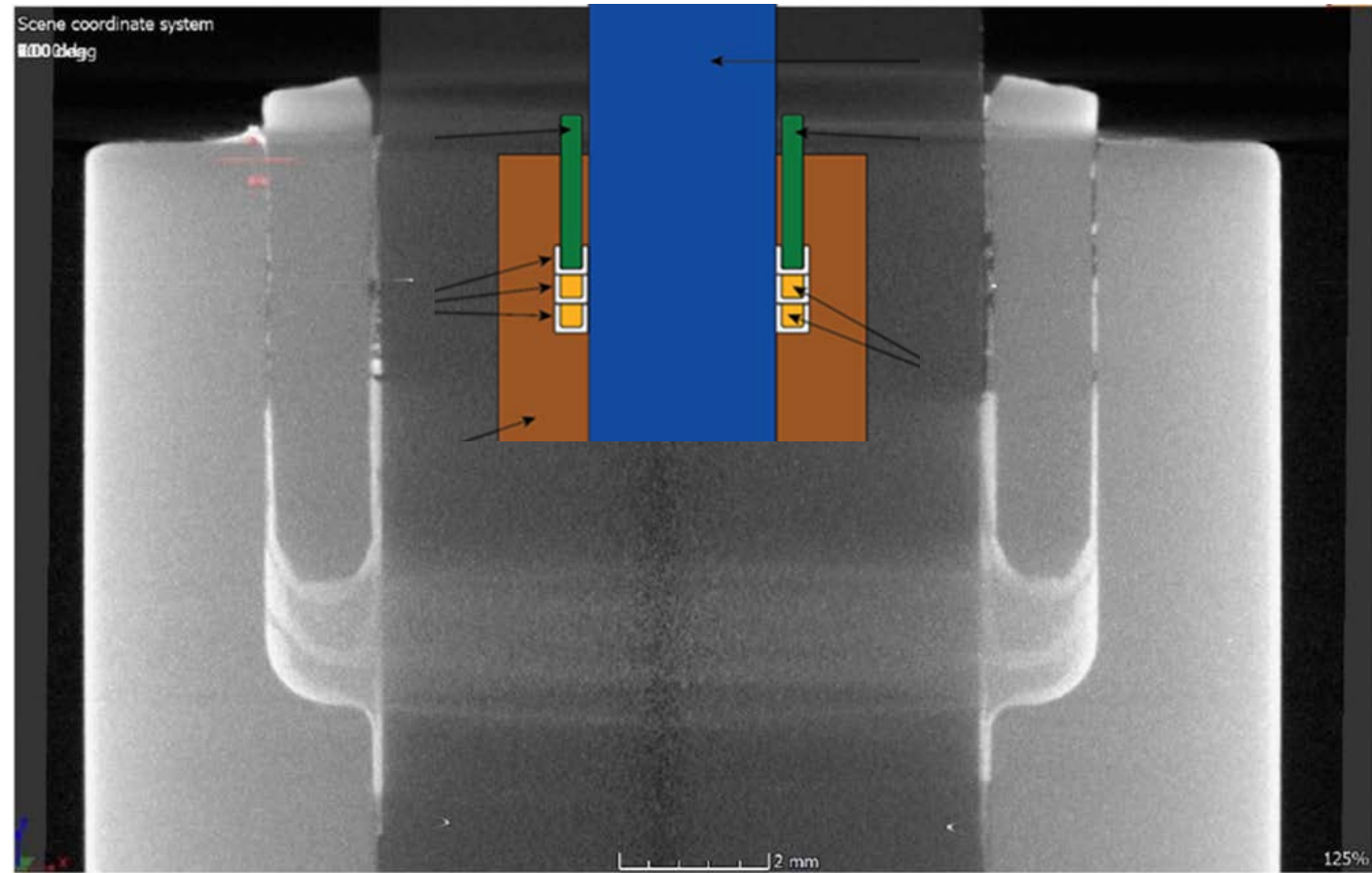
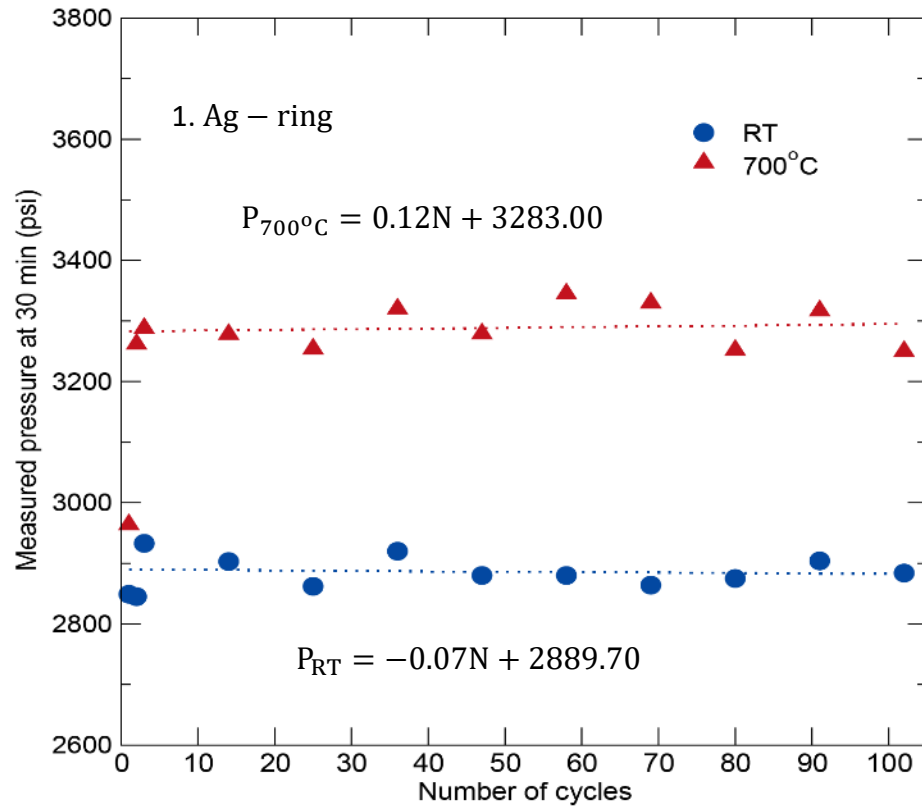


1. Richard Skifton at Idaho National Laboratory (INL)

2018 UTSR Project Review Meeting

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# Performance of Ag U-Ring Seal Assembly at High T and P

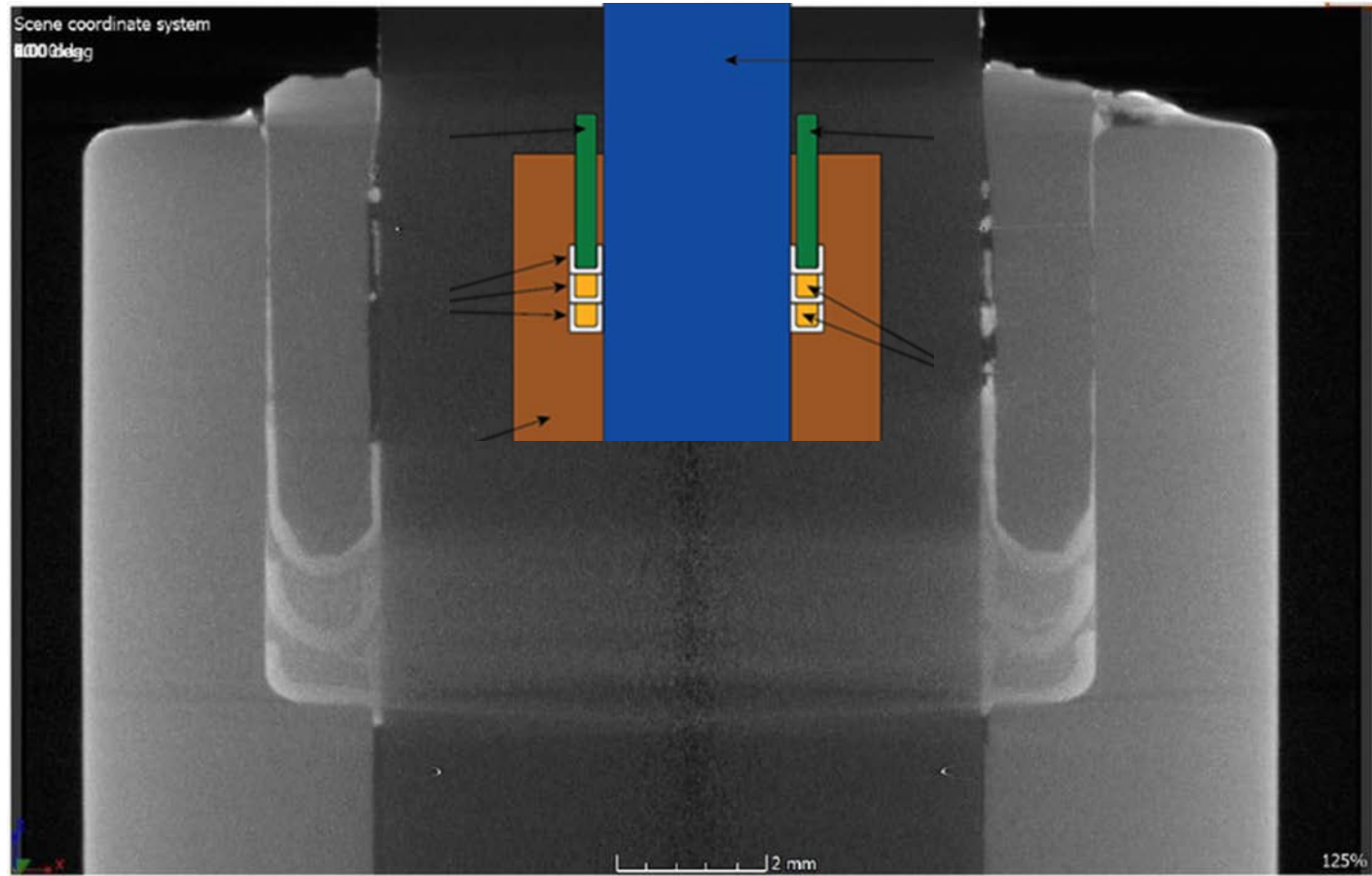
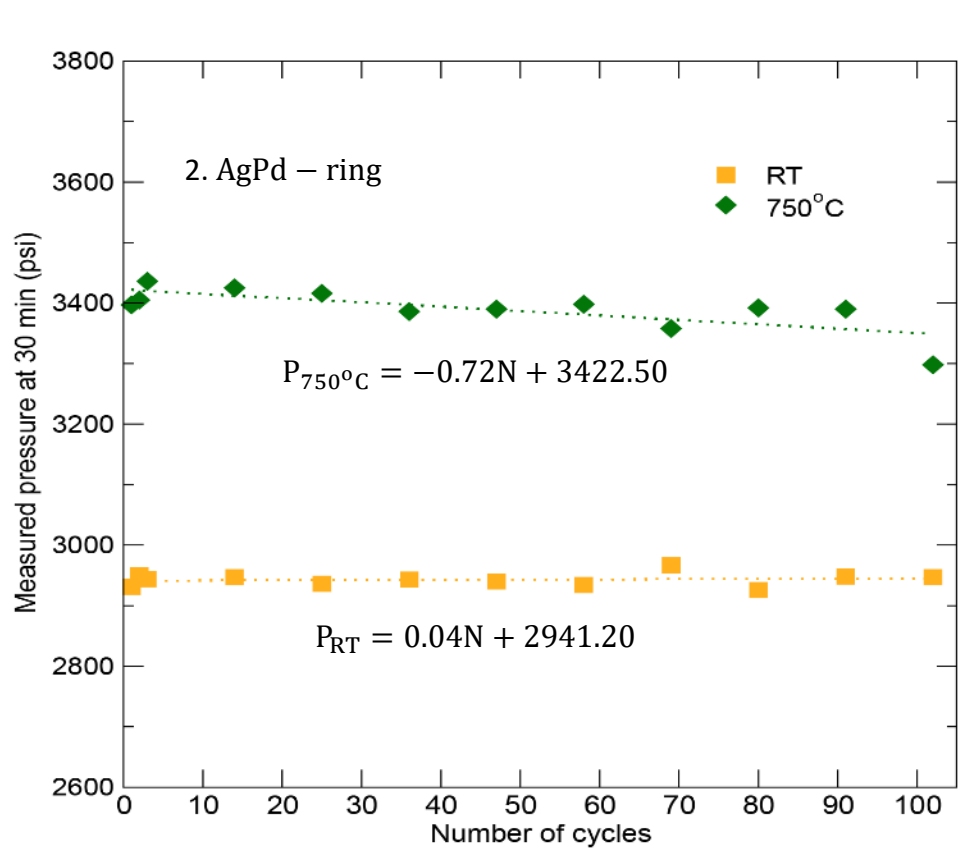


No.	Name	Tube	U-ring	Cermet	Ink
1	KH041018JOIN	Inconel 600	Ag	Ag75-MgO25	Ag
2	KH061318JOIN	Inconel 600	Ag75-Pd25	Ag75-MgO25	Ag

P = measured pressure (psi)  
N = number of cycles



# Performance of AgPd U-Ring Seal Assembly at High T and P

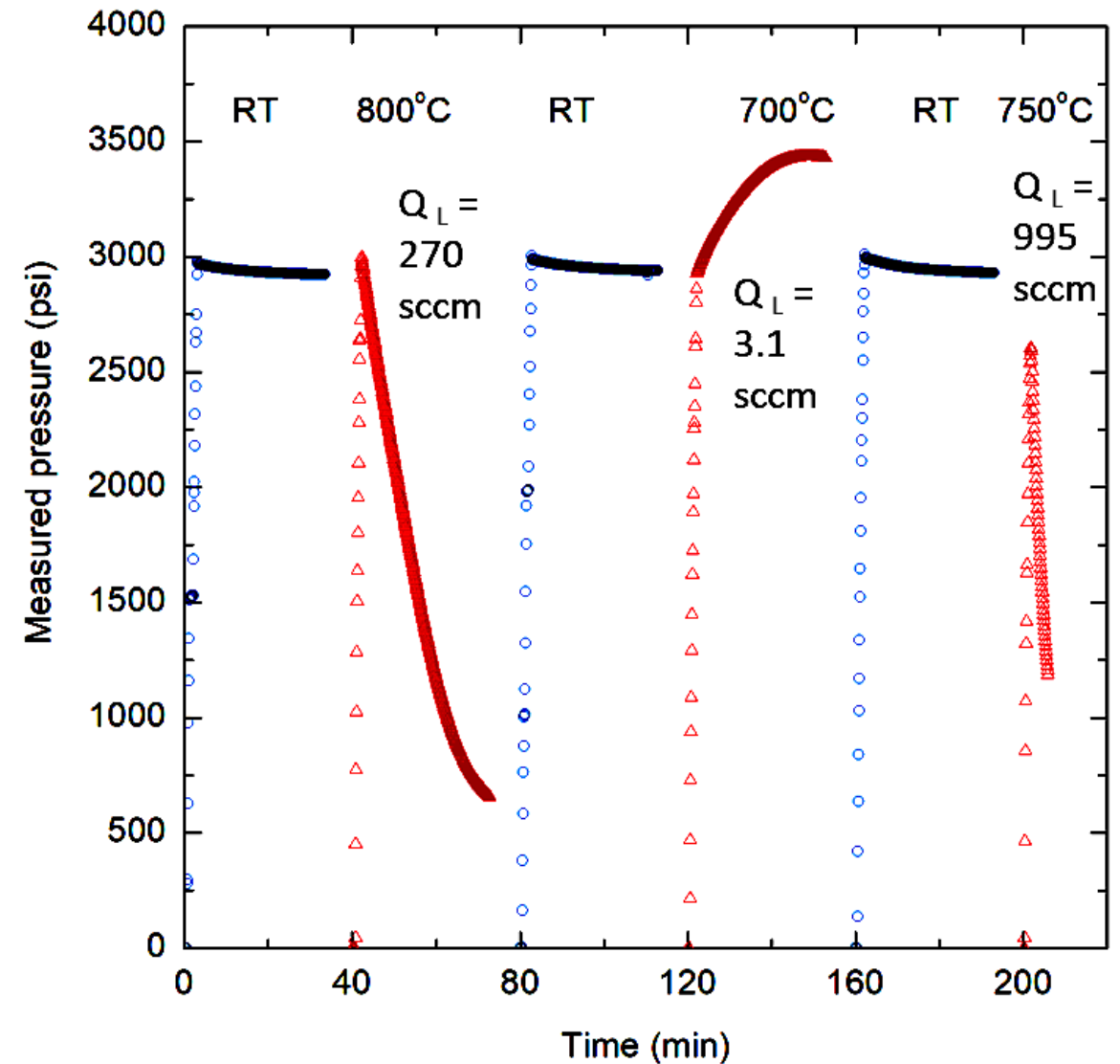


No.	Name	Tube	U-ring	Cermet	Ink
1	KH041018JOIN	Inconel 600	Ag	Ag75-MgO25	Ag
2	KH061318JOIN	Inconel 600	Ag75-Pd25	Ag75-MgO25	Ag

P = measured pressure (psi)  
N = number of cycles

# U-Ring Seal Assembly Performance at Higher Temperatures

- Ag U-Ring seal assembly leaked after single exposure to 760°C
- Ag (75%)-Pd (25%) alloy (AgPd) U-Ring performed well for >80 cycles at 750°C
- AgPd seal assembly failed after 1<sup>st</sup> thermal cycle to 800 °C





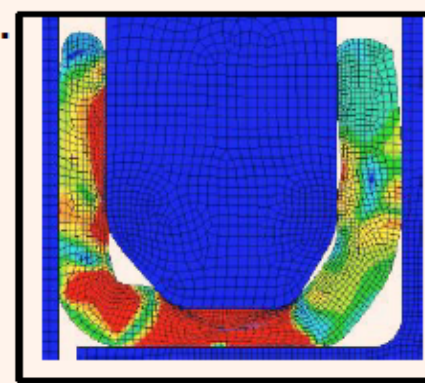
# FEA Modeling

- CAD and FEA modeling are used extensively on project to support multiple activities
  - Failure analyses and XRCT images
  - Tolerance stackup analyses
  - Hot seal forming simulation
    - Single U-Ring + top hat (2)
    - 3 U-rings + top hat (3)
    - Cermet compression (4)
    - Complete seal assembly (6)
  - U ring forming from Ag sheet stock (5)
  - Evaluation of alternate seal designs

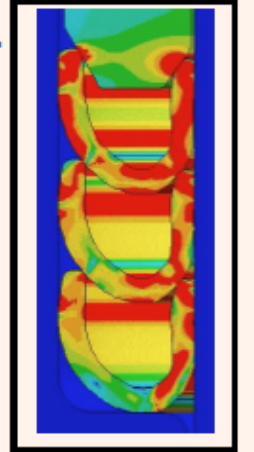
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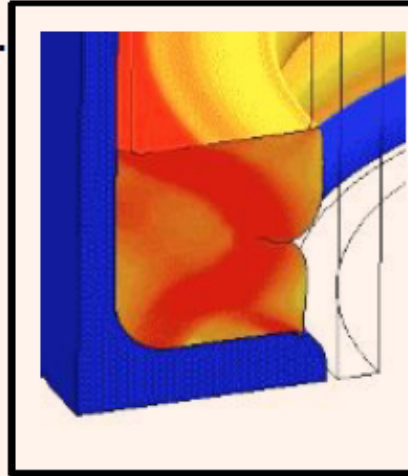
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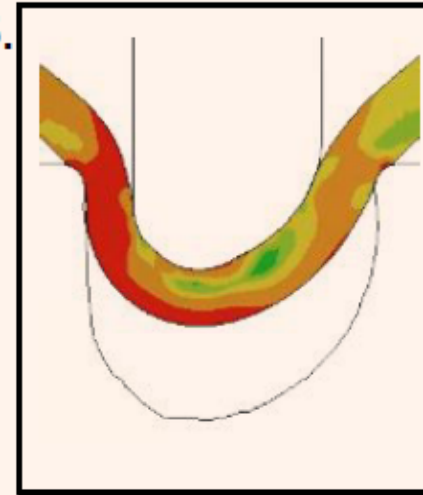
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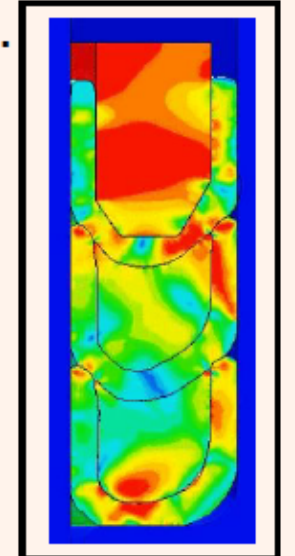
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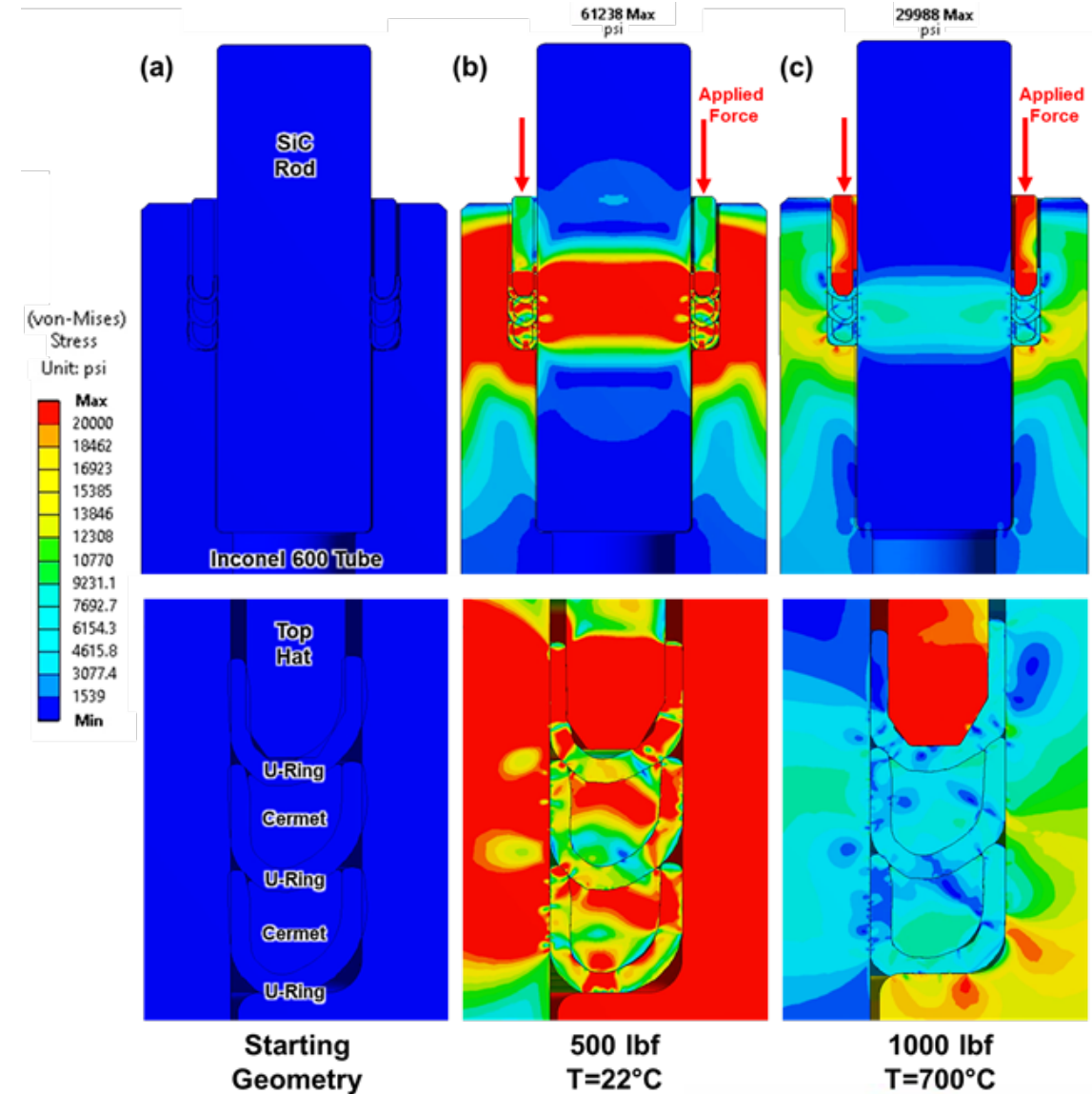


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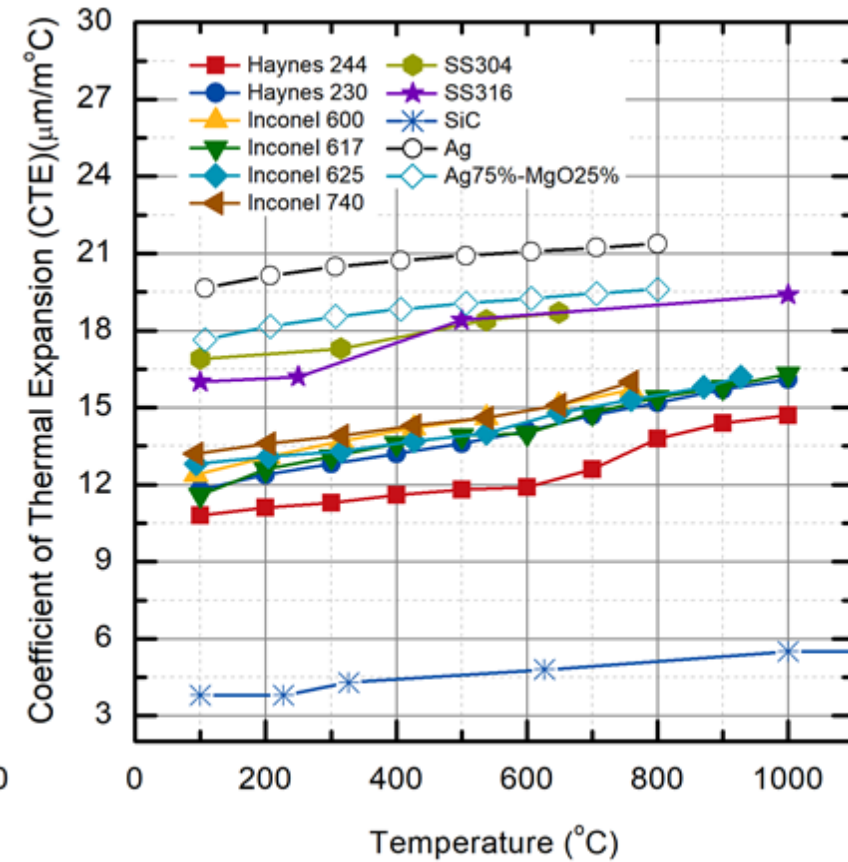
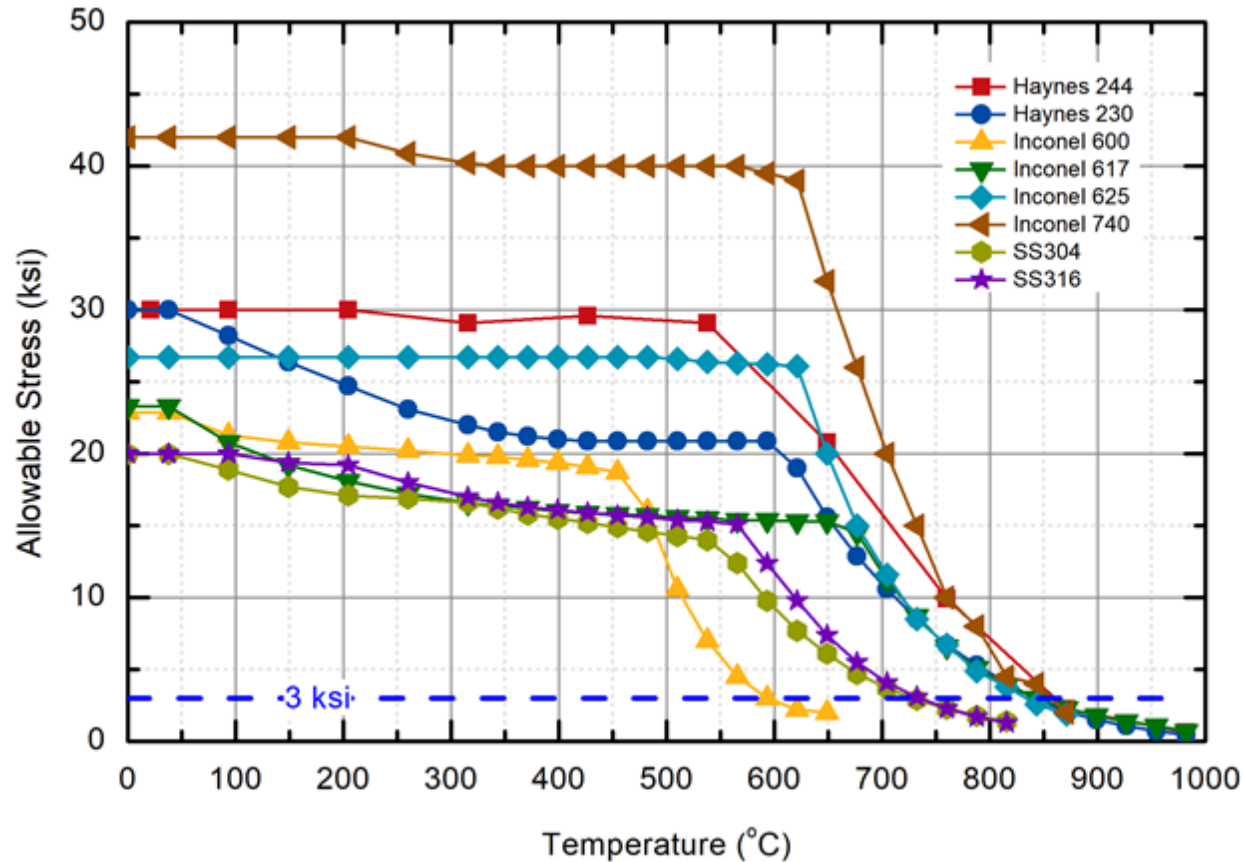
# FEA Modeling: Hot Seal Forming Process

- Developed FEA model of full seal assembly
- Using FEA model to evaluate influence of:
  - Temperature
  - Pressure
  - Ag vs. AgPd U-Ring, ink, and cermet
  - Geometric design optimization (part dimensions, radii, tolerances, etc.)
  - Metal alloy
  - Other design concepts



# Improvements for Higher Temperature Seals

- Allowable stress and CTE for high-temperature metal tubing narrows materials selection
- Use FEA models to optimize design and process reducing testing time and cost
- Verify machining tolerances
- Use XRCT to confirm baseline seal assemblies



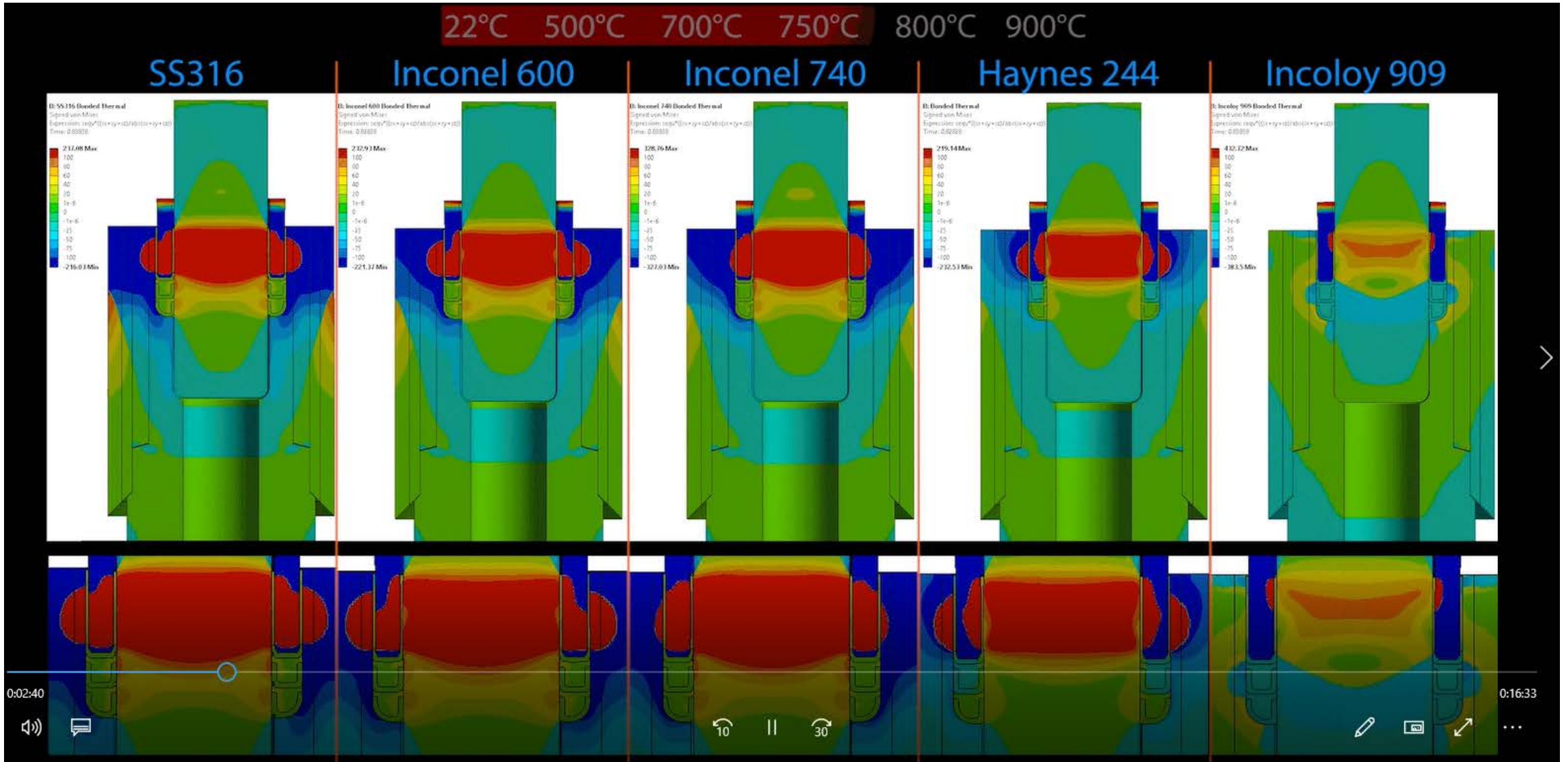
[1] ASME, ASME Boiler and Pressure Vessel Code: Section II Part D. New York, NY: ASME, 2015.

[2] Allowable stress and CTE were calculated from "Haynes 244 Alloy," Haynes International, Kokomo, IN, 2018.

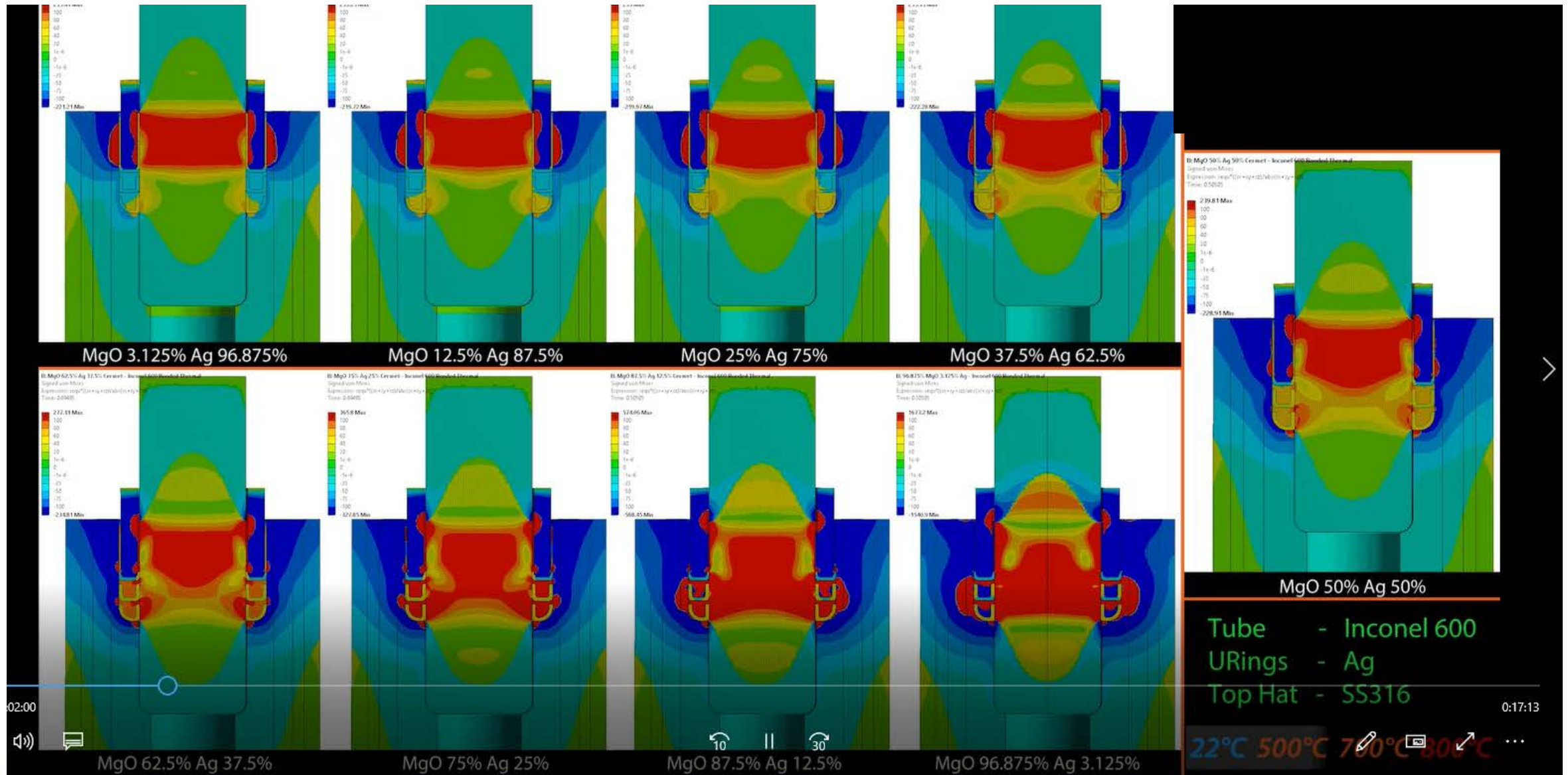
[3] Boise State University measured the CTE of SiC, Ag, and Ag75%-MgO25%.



# FEA Modeling: Influence of Tube Alloy and T



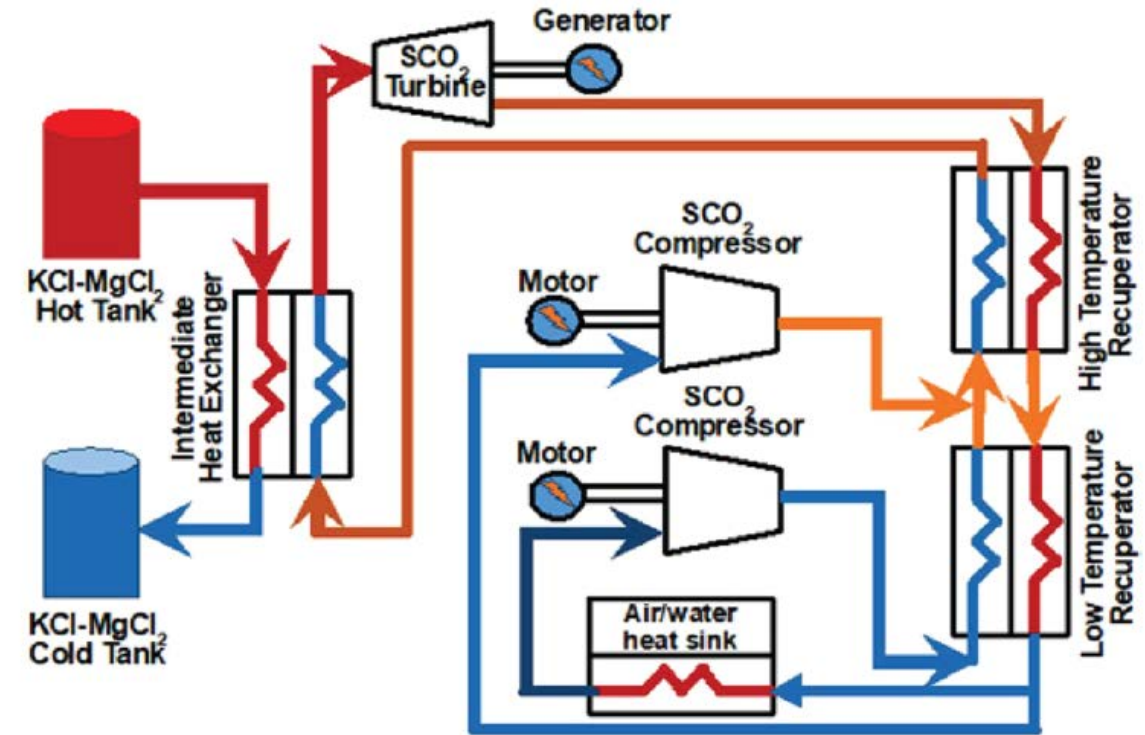
# FEA Modeling: Influence of Cermet Composition and T





# Voice of Customer (VOC) Inputs

- Need voice of customer (VOC) inputs to establish next demonstration prototype seal design
  - 1) Identify end users
  - 2) Identify applications
  - 3) Maximum seal temperature
  - 4) Maximum seal pressure
  - 5) Pressurization direction
  - 6) No. of thermal cycles
  - 7) Permissible leak rate
  - 8) Heat transfer fluids
  - 9) Tube size and material
  - 10) Heat exchanger type, material, and flows
  - 11) Access, footprint, logistics
  - 12) Cost
- Leverage high P and high T capabilities at DOE labs and/or potential commercialization partners for abuse/reliability testing of baseline and prototype seal designs



Caccia, M., et al. "Ceramic-metal composites for heat exchangers in concentrated solar power plants." *Nature* 562, no. 7727 (2018): 406.

# Summary

- Cermet-filled U-Ring seal design has demonstrated good performance
  - IN600/**Ag**/SiC seal at 3000 psi and **700 °C**
  - IN600/**AgPd**/SiC seal 3000 psi and **750 °C**
- IN600/AgPd/SiC seal failed after 1<sup>st</sup> thermal cycle to 800 °C
- May be related to fundamental material properties of metal tubing
- Evaluating the use of more exotic alloys to extend seal temperature limit
- Established XRCT capability in collaboration with INL for NDE of seal assemblies
- Initiated failure analysis (FA) plans to identify root cause(s), corrective action(s), and refine seal design and forming process
  - XRCT NDE
  - Destructive cross-sections, SEM/EDS
  - FEA coupled with XRCT and FA for supporting forming process improvements
- Initiated efforts to get VOC requirements

# Future Efforts

- Fabricate and test more Ag alloy-based seals
- Continue to perform detailed failure analysis on failed and baseline seals
- Continue to use FEA and FA coupled with experiment results to optimize hot seal forming conditions for Ag Alloy U-Rings, paste, and cermet
- Continue cermet characterization and diffusion bonding first principles studies in parallel and incorporate results as appropriate
- Obtain VOC inputs for high T seal applications for advanced power generation: seal operating conditions, fluids, T, P, dimensions
- Leverage VOC inputs to identify applications that may be able to use existing and improved seal designs
- Use FEA and FA tools to develop new designs that may be able to overcome the metal tubing temperature limitations
- Benchmark high P and high T testing capabilities at DOE labs and/or potential commercialization partners